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European Polymer Journal

journal homepage: www.elsevier.com/locate/europolj

Feature Article

Thermally degradable thermosetting materials

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ARTICLE INFO

Article history:

Available online 29 September 2010

Dedicated to Professor Nikos Hadjichristidis in recognition of his contribution to polymer science.

Keywords:

Re-workable

Degradable linkage

Thermosetting materials

Adhesive

ABSTRACT

Thermosetting materials have been widely used in a variety of applications but they generally display poor tractability after curing, which limits their use in applications where degradable or re-workable polymers are advantageous. Moreover, recyclability and biodegradability of thermosetting polymer also limit their use in applications where recycling and biodegradation are important. A variety of thermally degradable linkages within thermosetting materials have been studied both in academia and industry to develop re-workable adhesives. This review reports the recent development in thermosetting materials containing thermally breakable linkages that exhibit re-workability as well as potential for recyclability and biodegradability.

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1. Introduction

Thermosetting materials have been widely used in a variety of applications such as coatings, encapsulants, and adhesives [1–3]. However, many traditional thermosetting materials display poor tractability which limits their use in applications where degradability or re-workability is advantageous. The use of an adhesive that will melt or decompose to allow removal of individual chips for repair or replacement would be an advantage for semiconductor manufacturers. In order to develop adhesives that are re-workable, thermally degradable linkages have been introduced into the crosslinkable structures. The decomposition of the degradable linkages upon heating is expected to lead to a decrease in crosslinking density and modulus of the adhesive, allowing chip removal and replacement. The optimal temperature range for safe re-work operation is desired to be within 200–250 °C. Moreover, recyclability and biodegradability of thermosetting polymer limit their use in applications where recycling and biodegradation are important [4,5]. A variety of degradable linkages within thermosetting

materials have been studied both in academia as well as in industry. This review reports the recent development in thermosetting materials containing thermally breakable linkages that exhibit re-workability as well as potential for recyclability and biodegradability.

2. Thermosetting materials based on epoxy

Epoxy resins are very important materials and are widely used in the field of electronic packaging because of their unique properties, such as excellent chemical and corrosion resistances, electric and physical properties, high adhesion, low shrinkage on curing, and good thermal stability [6]. However, the resulting networks permanently affix the components. Although, the interactability of these materials is desirable from reliability aspect, it is undesirable from a re-workability or recyclability point of view. Several research groups have reported re-workable epoxy-based thermosetting materials with incorporating thermally cleavable linkages into their monomers.

2.1. Thermosetting epoxy materials with ester linkages

The synthesis and characterisation of a series of cycloaliphatic epoxy compounds that incorporate ester groups

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has been reported [7,8]. The epoxy compounds either contain secondary or tertiary esters linking the epoxy groups or form tertiary ester bonds during the curing reaction, **1–5**, Fig. 1. The secondary and tertiary esters were identified as potential heat-cleavable linking groups that can decompose with or without added acid, based on thermal decomposition mechanisms of esters [9]. The cycloaliphatic epoxy monomers **1–5** were derived from the esterification of cyclohexenoic acid with terpineol followed by epoxidation.

The epoxy monomers were cured by hexahydro-4-methylphthalic anhydride (HHMPA) as hardener and 1-cyanoethyl-2-ethyl-4-methylimidazole (2E4MZ-CN) as catalyst.

The thermal decomposition study of the resulting thermosetting materials with ester linkages showed that they start decomposing at temperature between 250 and 300 °C, whereas the conventional epoxy-based thermosetting material which contain primary ester bonds did not decompose until the temperature reached 350 °C. The breakdown of the thermosetting materials esters was also followed through their Glass Transition temperature (T_g) and by Fracture toughness tests. During the degradation test, the crosslink density, strength and T_g decreased simultaneously.

However, aliphatic esters are usually vulnerable to hydrolysis and the resistance to hydrolysis is critical for the electronic packaging. This led to the synthesis and characterisation of two isophthalate-type di-epoxides; one with two secondary ester linkages, **6**, and the other with two tertiary ester linkages, **7**, Fig. 1 [10]. The reaction of tetrahydrobenzaldehyde with methyl magnesium bromide in THF gave cyclohexenyl ethanol which was treated with isophthaloyl dichloride in the presence of pyridine to give bis(cyclohexenyl ethyl) isophthalate. The subsequent

epoxidation reaction led to the formation of **6**. Reaction of isophthaloyl dichloride with terpineol gave bis[methyl-(methylcyclohexenyl)ethyl] isophthalate which was epoxidised to give **7**.

The di-epoxide monomers were similarly cured by HHMPA and 2E4MZ-CN as hardener and catalyst, respectively. The thermal stability of thermosetting materials from cured **6** and **7** was studied with TGA. The result showed that thermosetting materials from cured **6** and **7** exhibited weight loss at 330 and 250 °C, respectively. The difference in the decomposition temperature was attributed to the presence of tertiary esters in thermosetting material from cured **7**.

2.2. Thermosetting epoxy materials with carbamate linkage

A series of cycloaliphatic di-epoxide monomers containing thermally cleavable carbamate linkages were synthesised and characterised, **8–13**, Fig. 2, [11]. Cyclohexene carbonyl chloride was reacted with sodium azide to form cyclohexene isocyanate which was then reacted with cyclohexene methanol to produce carbamate containing two cyclohexene groups. The epoxidation of cyclohexene compounds produced di-epoxide monomers containing carbamate.

The di-epoxide monomers containing carbamate linkages were also cured by HHMPA as hardener and 2E4MZ-CN as catalyst. The thermosetting materials from cured monomers **8–13** started to decompose at 250–300 °C as compared with 350 °C for the cured sample of the commercial cycloaliphatic epoxide without carbamate linkages. However, the changes that occurred in the epoxy network during the re-work process are presently poorly understood. Moreover, the system is further complicated

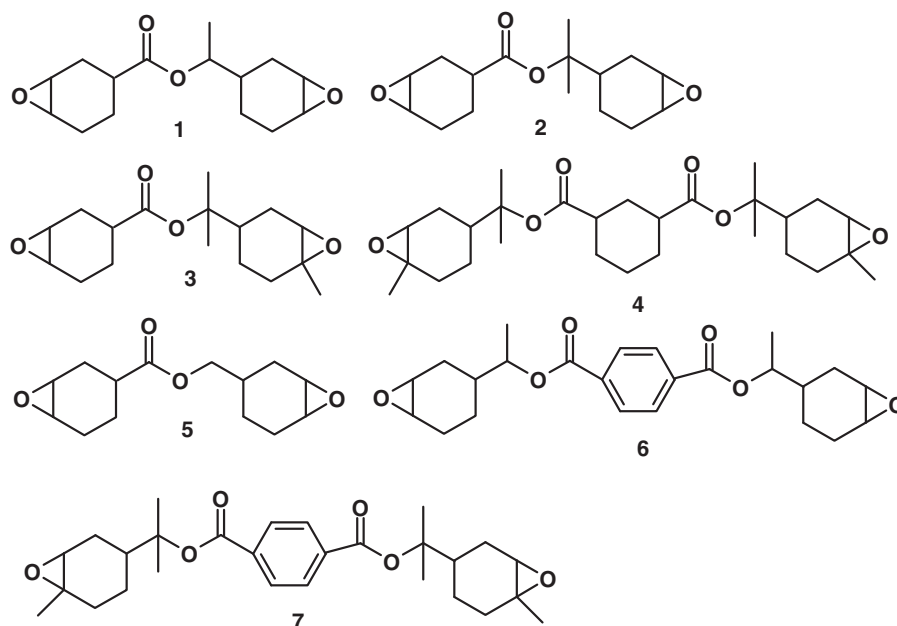


Fig. 1. Di-epoxide monomers **1–7** containing ester linkages.

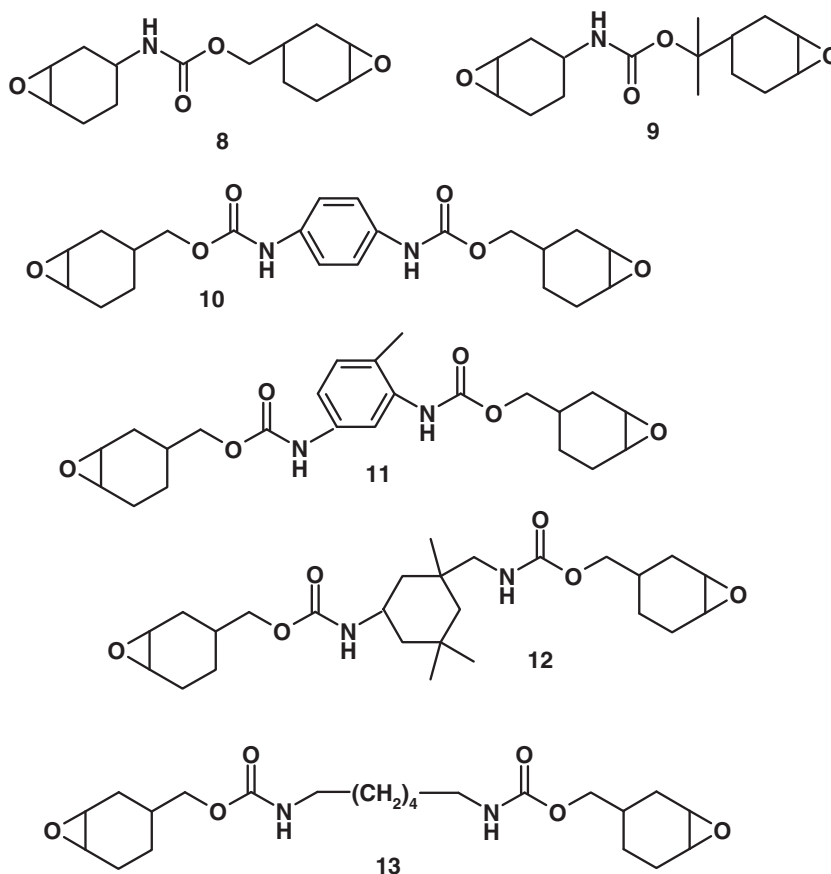


Fig. 2. Di-epoxide monomers 8–13 containing carbamate linkages.

by the fact that the carbamate groups within the di-epoxides can act as an internal catalyst.

2.3. Thermosetting epoxy materials with carbonate linkages

The synthesis of four cycloaliphatic or aliphatic epoxide monomers containing carbonate linkages, has been reported, **14–17**, Fig. 3 [12]. Cycloaliphatic di-epoxides monomers **14** and **15** contain primary and secondary carbonate linkage, respectively. Monomer **16** is a cycloali-

phatic mono-epoxide containing a tertiary carbonate group, whereas monomer **17** is a di-epoxide with cycloaliphatic and aliphatic epoxides on each end and it contains one tertiary carbonate linkage. These monomers were synthesised by using triphosgene to convert alcohol or phenol into chloroformate which were then reacted with terpineol to form carbonate. The epoxidation of cyclohexene compounds produced the desired epoxide containing carbonate linkages. Although, the synthesis of primary or secondary carbonates with aliphatic groups on both sides

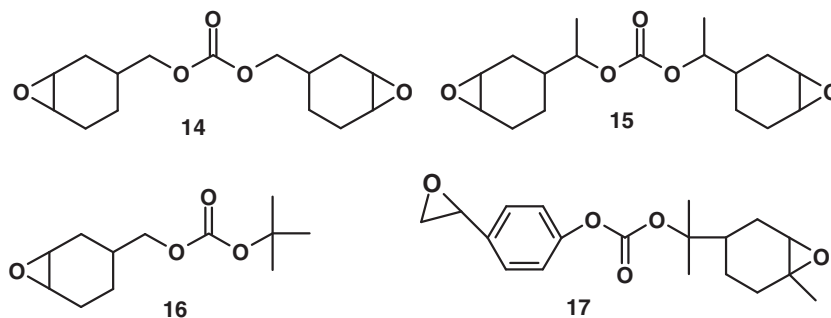


Fig. 3. Di-epoxide monomers 14–17 containing carbonate linkages.

were reported to be straightforward, but a carbonate containing tertiary aliphatic groups on one or two sides was much more difficult to prepare.

The epoxide monomers **14–17** containing carbonate linkages were cured by HHMPA as hardener and 2E4MZ-CN as catalyst. The thermosetting materials with carbonate linkages were shown to start to decompose at temperature lower than 350 °C, the decomposition temperature for the cured sample of the commercial cycloaliphatic di-epoxide.

2.4. Thermosetting materials based on maleimides

The decomposition temperatures of the cured epoxy resin containing esters, carbamates and carbonates linkages reported above are very close to that of epoxy resin without these linkages of 350 °C. Furthermore, these degradable thermosetting materials are based on traditional epoxy resin.

Thermosetting adhesives based on maleimides have been developed for electronic packaging applications. These systems are advantageous due to their fast cure speed as well as other material specific advantages. For example, maleimide-based materials [13–16] have been found to exhibit better adhesion and low shrinkage after curing. In addition, maleimide functionality can be homopolymerised and also copolymerised efficiently with a variety of comonomers, such as styrenic, vinyl ether, acrylate, and methacrylate by free radical processes. However, these systems are intractable after curing and therefore not suitable for applications where adhesive removal is desired, such as de-bonding and re-workable adhesives. Therefore, attempts have been made to develop thermosetting materials based on maleimide compounds that can provide the advantages offered by maleimide adhesives in a re-workable adhesive system.

2.4.1. Thermosetting materials based on maleimides containing acetal ester linkages

Recently, re-workable thermosetting materials based on maleimide were prepared by introducing degradable acetal ester linkages into multi-functional maleimide monomers [17]. Five monomers containing acetal ester linkages and maleimide functionality were synthesised, **18–22**, Fig. 4. Neat reaction of maleimidocaproic acid with 1,4-butanediol divinyl ether, cyclohexanedimethanol divinyl ether, diethyleneglycol divinyl ether and cyclohexyl vinyl ether gave di-functional maleimide monomers **18**, **19**, **20** and **21**, respectively, containing acetal ester groups. The reaction of maleimidopropionic acid with diethyleneglycol divinyl ether gave mono-functional maleimide monomer **22**. The Di-functional maleimide monomer without acetal ester group **23** was also synthesised to prepare thermosetting material to provide basis for comparison [18].

These multi-functional maleimide monomers were polymerised via free radical polymerisation process initiated by 1,1-di(*tert*-amyl peroxy) cyclohexane (USP-90MD) [19] to obtain thermosetting materials containing acetal ester linkages. The thermal degradation behaviour of the thermosetting materials was analysed by DSC, TGA, GC–MS, and hot-stage FTIR.

It was found that the acetal ester linkage degraded rapidly above 225 °C and introduction of this linkage into the adhesive formulation led to improved re-workability of adhesively bonded substrates. The TGA study of the thermosetting materials from cured monomer **18** showed that the material displayed 10% weight loss at 242 °C, Fig. 5. In contrast thermosetting materials from compound **23** without acetal ester linkage displayed 5% weight loss at 300 °C, Fig. 6.

The re-work test was performed by testing the adhesion of the polymerised materials in an electronic package at

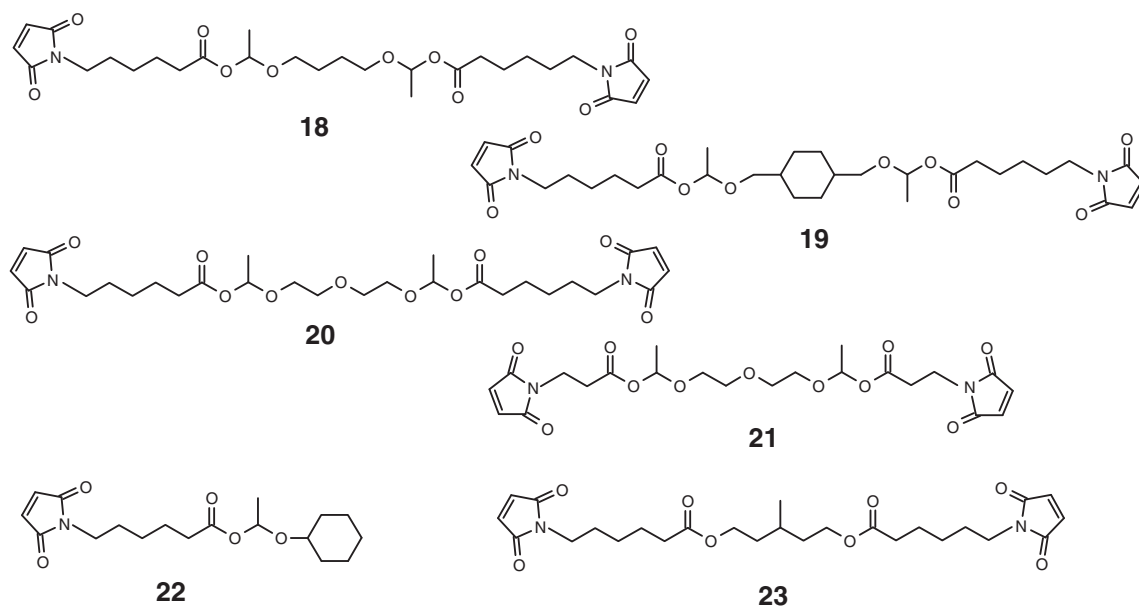


Fig. 4. Structures of maleimide based monomers **18–23** containing acetal ester groups.

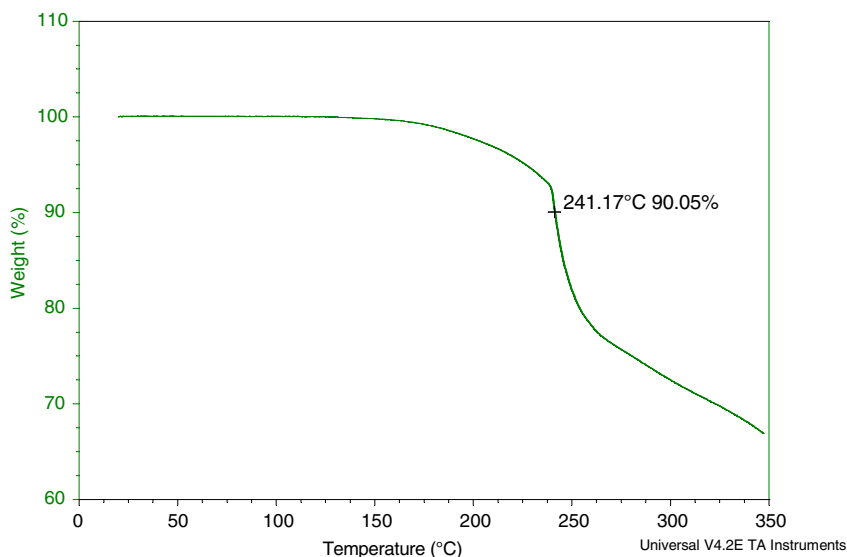


Fig. 5. TGA thermogram for thermosetting material from curing monomer **18**.

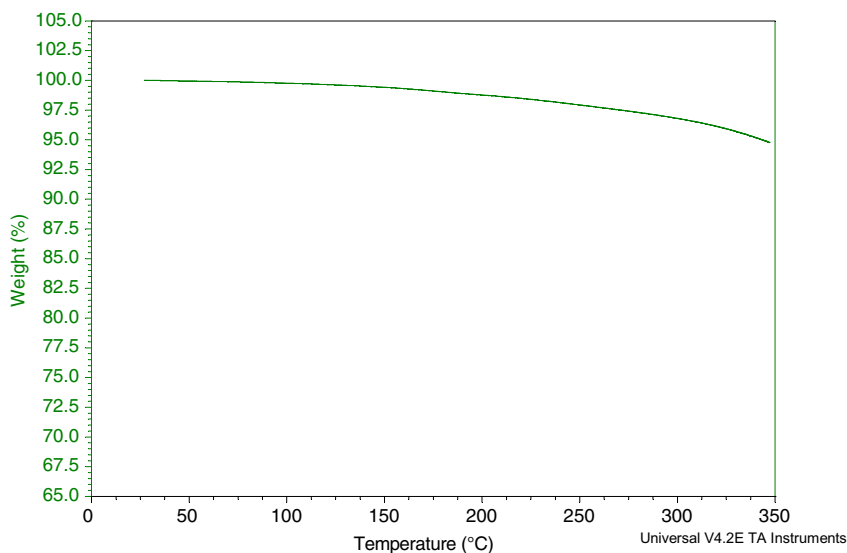


Fig. 6. TGA thermogram for thermosetting material from curing monomer **23**.

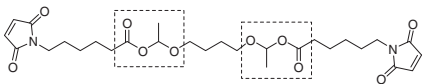
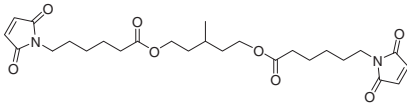
high temperature. The mixture of compound **18** and USP-90MD was used as underfill [20] in a standard electronic package, followed by curing at 120 °C for 10 min. The adhesion of the cured underfill was tested by die shear test at both ambient temperature and 150 °C [21]. Monomer **23**, without acetal ester linkage, was also tested as a reference molecule. The adhesion results are summarised in Table 1. The adhesion strength of cured compound **18** dropped significantly from 35.2 kg (room temperature) to 2.3 kg (150 °C) after it was held at 250 °C for 2 min. However, the adhesion of cured monomer **23** was much higher after 250 °C exposure. In addition, board damage was observed for cured compound **23** during the die shear

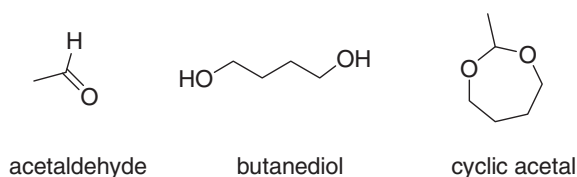
test due to cohesive failure of the substrate, demonstrating poor re-workability. The die shear data suggests that the acetal ester linkage indeed caused a large decrease in adhesion strength after exposing to elevated temperature, which facilitates the re-work process.

Hot-stage FTIR of the thermosetting material from cured monomer **18** showed that at ~250 °C, the intensity of the acetal C–O peak at ~1135 cm^{−1} dropped in intensity indicating that the observed breakdown in network structure is caused by the degradation of the acetal ester linkages.

Thermal desorption GC–MS was conducted on the thermosetting material from cured monomer **18** to analyse

Table 1Die shear adhesion of cured **18** and **23**.

Thermosetting Material	25 °C adhesion	150 °C adhesion (after exposing to 250 °C for 2 min)
 <p style="text-align: center;">18</p>	35.2 kg	2.3 kg
 <p style="text-align: center;">23</p>	44.9 kg	18.6 kg

**Fig. 7.** Structures of the decomposition products at 250 °C.

the degradation products. After heating at 250 °C for 10 min, volatilised components were collected and analyzed by GC–MS. The main decomposition product was found to be a cyclic acetal but small amounts of acetaldehyde and butanediol were also present, [Fig. 7](#).

2.5. Thermosetting ROMP materials

Ring opening metathesis polymerisation (ROMP) initiated by Grubbs well-defined ruthenium initiators has been shown to result in the synthesis of well-defined polymers with controlled architectures, molecular weights, polydispersities, and terminal functionalities [22–26]. A ROMP processing technique using Grubbs well-defined ruthenium initiators has also been developed for the synthesis of thermosetting materials with well-defined cross-linked networks from mixtures of mono- and di-functional norbornene dicarboximide monomers for applications in Resin Transfer Moulding (RTM) and Reaction Injection Moulding (RIM) [27,28]. An excellent level of control over crosslink density has been demonstrated by controlling the amount of added di-functional monomer to the reaction mixture. However, these ROMP thermosetting materials were shown to be thermally stable, degrading above 400 °C, and could be used for applications where high temperature stability is required.

2.5.1. Thermosetting ROMP materials containing acetal ester linkages

Recently, research has been conducted to render the well-defined thermosetting ROMP materials thermally degradable, biodegradable and recyclable by introducing degradable linkages into the crosslinkable structures [29].

The benefit of developing recyclable and biodegradable thermosetting materials in an increasingly environmentally conscience society is obvious as it will render the thermosetting materials environmentally friendly [30].

The decomposition temperatures of the epoxy-based thermosetting materials containing esters, carbamates and carbonates linkages reported earlier are very close to that of epoxy resin without these linkages of 350 °C. Moreover, the thermosetting materials containing esters, carbamates, carbonates and acetal esters exhibit 100% weight loss at temperature between 250 and 350 °C since these linkages are incorporated into the main chains. Therefore, well-defined thermosetting materials containing acetal ester linkages have been prepared by an efficient, controllable and reproducible process at room temperature such as ROMP. The crucial part of the design of these thermosetting materials was the thermal breakdown of only the linkages, leaving the main chains intact allowing the transition from thermosetting to thermoplastic facilitating the recycling. The system reported was based on the synthesis of mono- and di-functional norbornene dicarboximide monomers containing acetal ester groups and their ROMP to produce well-defined thermosetting materials.

Mono- and di-functional norbornene dicarboximide monomers **24** and **25**, containing acetal ester groups were synthesised, [Fig. 8](#). The reaction between *exo*-norbornene dicarboxylic anhydride and 6-aminocaproic acid in refluxing toluene gave *N*-caproic acid-*exo*-norbornene dicarboximide. The reaction of *N*-caproic acid-*exo*-norbornene dicarboximide with and butyl vinyl ether and 1,4-butanediol divinyl ether gave the mono-functional monomer **24** and the di-functional monomer **25**, respectively, both containing the acetal ester group. The structures of monomers **24** and **25** were confirmed by NMR Spectroscopy, mass spectroscopy and elemental analysis. Furthermore, FT-IR spectra of monomers **24** and **25** clearly showed the presence of acetal C–O bonds at 1135 cm^{−1}.

The di-functional monomer **25** was subjected to ROMP using Grubbs ruthenium first generation initiator to produce the thermosetting material **26**. Mixtures of di-functional **25** and mono-functional **24** monomers were also

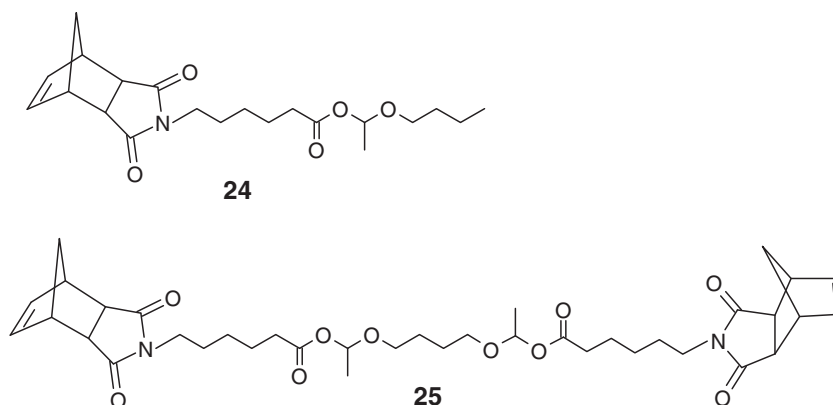


Fig. 8. Mono- and di-functional norbornene dicarboximide monomers **24** and **25** containing acetal ester linkage.

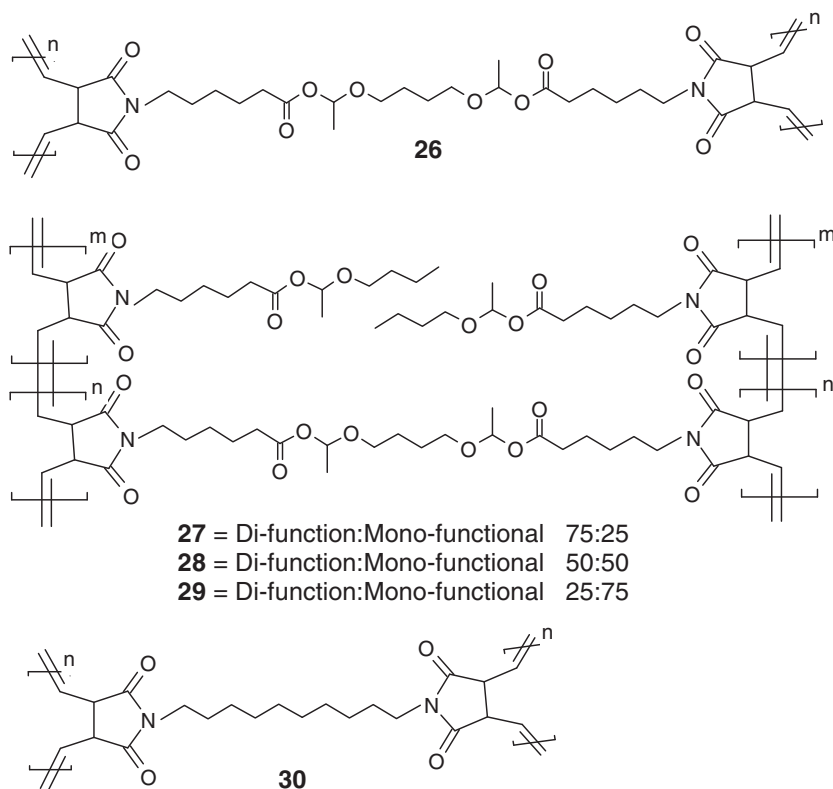


Fig. 9. Thermosetting materials **26–30**.

subjected to ROMP using Grubbs ruthenium first generation initiator to produce thermosetting materials with different degrees of crosslinking. Three thermosetting materials **27–29** were prepared using molar ratios of di-functional:mono-functional monomer of 75:25; 50:50, and 25:75, respectively. Fig. 9 shows idealised structures for thermosetting materials **26–29**, but the network was also reported to contain some di-functional units incorporated through only one norbornene moiety [28]. FT-IR spectra of the thermosetting materials showed the presence of an acetal C–O band at 1135 cm^{-1} .

The thermal degradation of the thermosetting material **26** studied by TGA showed that the material was stable to $150\text{ }^{\circ}\text{C}$ but exhibited weight loss of 14%, 30% and 45% at $200\text{ }^{\circ}\text{C}$, $250\text{ }^{\circ}\text{C}$ and $300\text{ }^{\circ}\text{C}$, respectively, Fig. 10. The thermal degradation of the materials **27–29** was also studied by TGA under the similar conditions to that for material **26**, Fig. 10. The weight loss was found to be 36%, 25% and 21%, respectively, for thermosetting materials **27**, **28** and **29** at $250\text{ }^{\circ}\text{C}$. This behaviour was reported to be consistent with thermosetting material **27** having the highest di-functional content, highest degree of crosslinking and hence

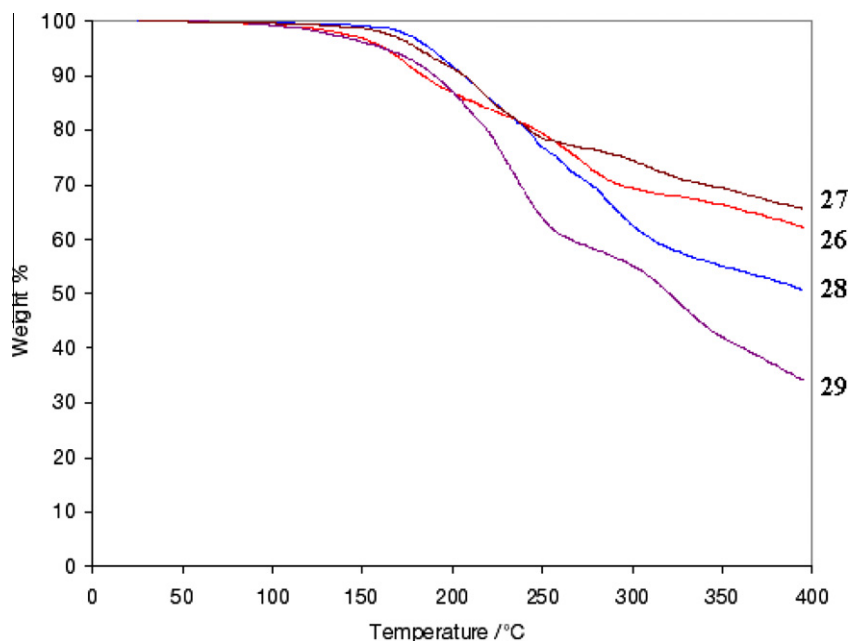


Fig. 10. TGA thermographs observed during the thermal degradation of materials **26–29**.

higher weight loss; and thermosetting material **29** having the lowest di-functional content, least degree of crosslinking and hence lower weight loss.

Thermal degradation studies on thermosetting ROMP material prepared from mono- and di-functional norbornene dicarboximide without acetal ester linkages **30** [28], showed that the thermosetting material was very stable to 400 °C, Fig. 11. Therefore, it was concluded that any fragments observed during TGA–MS analysis must be the result of the thermal decomposition of the acetal ester linkage which was supported by considerable reductions in the intensity of C–O bands at 1135 cm^{-1} in the FT-IR spectra observed for the retrieved materials. Materials **26–29** were subjected to TGA–MS analysis to determine the fragments resulted from thermal decomposition of

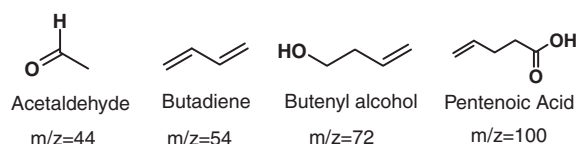


Fig. 12. Fragments and their corresponding structures.

the materials by taking sequential mass spectrometric traces at 50 °C intervals.

The most abundant fragments detected for the degradation of the material **26–29** were between 200 and 250 °C, possessed m/z ratios of 44, 54, 72 and 100 mass units. The fragments with m/z ratios of 44, 54, 72, and 100 units were believed to be corresponding to acetaldehyde, butadiene, butenyl alcohol and pentenoic acid, respectively, Fig. 12.

Interestingly, these thermosetting ROMP materials containing acetal ester linkages were shown to be breaking down around 200 °C, which is in the same region as the lead-free solder reflow temperature (217 °C). This is extremely advantageous in the electronic industry as it would allow easy chip removal and replacement, if these materials are used as adhesives.

3. Conclusions

Thermosetting materials based on epoxy containing ester, carbamate and carbonate linkages were synthesised and characterised. The decomposition temperatures of the cured epoxy resin containing esters, carbamates and carbonates linkages reported above are very close to that of epoxy resin without these linkages of 350 °C.

Thermosetting materials based on maleimides containing acetal ester linkages have also been synthesised via a

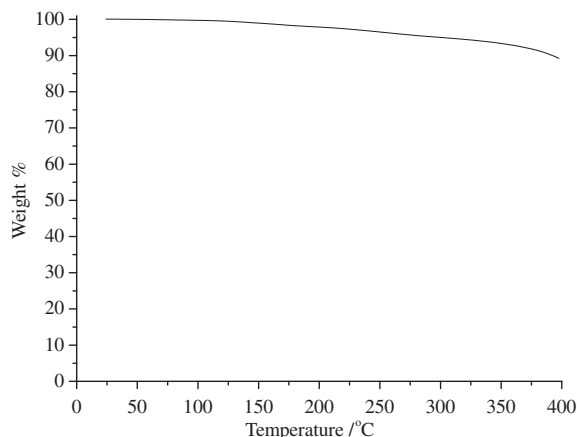


Fig. 11. TGA thermographs for the thermal degradation of thermosetting material without acetal ester linkages **30**.

free radical polymerisation process. The thermosetting materials started to degrade $\sim 225^\circ\text{C}$ and reached a maximum at $\sim 250^\circ\text{C}$, due to the thermal breakdown of the acetal ester groups. The thermosetting material showed good re-workability.

Well-defined ROMP thermosetting materials based polymerisation of either di-functional norbornene dicarboximide monomers or a mixture of mono- and di-functional norbornene dicarboximide monomers all containing acetal ester groups have recently been synthesised. The TGA study of the resulting thermosetting materials showed that they are stable to 150°C and that they exhibit rapid breakdown at about 200°C , as the result of the thermal decomposition of the acetal ester linkage. The breakdown temperature of 200°C is in the same region as the lead-free solder reflow temperature (217°C) which is advantageous in the electronic industry as it would allow easy chip removal and replacement, if these thermosetting materials are used as adhesives.

The thermosetting materials containing esters, carbamates, carbonates and acetal esters exhibit 100% weight loss at temperature between 250 and 350°C since these linkages are incorporated into the main chains. In contrast, the well-defined ROMP thermosetting materials only showed partial decomposition due to the breakdown of the acetal ester groups within the crosslinking units and the backbone chains remained stable to over 400°C . This is expected to allow transition from thermosetting to thermoplastic materials and hence facilitate the recycling of the thermosetting materials.

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Ezat Khosravi (born 1950) obtained his doctorate in Polymer Science from the University of Sussex, UK in 1981. He joined Prof. N.C. Billingham as a Postdoctoral Research Assistant, School of Molecular Sciences, Sussex University, in 1982. He joined the groups of Professor W. J. Feast and Professor V. C. Gibson at Chemistry Department, Durham University, UK in 1987 as a Research Assistant. He then joined the Interdisciplinary Research Centre in Polymer Science and Technology at Durham University in 1990 to work with

Professor Feast as a Senior Research Fellow. He was promoted to a Reader in Chemistry in 2001 at the Chemistry Department, Durham University. Research interests range from well-defined polymerisation chemistry to novel polymers and biopolymers. One of the current projects is focusing on the synthesis of well-defined and degradable thermosetting materials by incorporating thermally degradable as well as biodegradable linkages into the crosslinkable structures.



Osama M. Musa (born 1968) earned two Master of Science degrees in Organic Chemistry (heterocyclic, 1992) and Polymer Chemistry in 1994, and a Doctorate in Organic Chemistry in 1997 from Wayne State University, Michigan, USA under Prof. Martin Newcomb, with whom he also completed a post doctoral fellowship. He joined National Starch & Chemical Company Bridgewater, New Jersey, USA in 1998 where he was the Reactive Chemistry and Polymer Modification Pillar Leader. He received The “CEO Innovation Award” in 2001, The “Corky” Award for contributions to “BMI Hybrid Chemistry”, The “BeSCCo” Innovation Award in 2003 and joined the National Starch’s “Inventors Hall of Fame” in 2004. He joined International Specialty Products (ISP) Corporation, New Jersey, USA in 2008 where he is now Senior Director of Molecular & Analytical Science Group. His current interests are novel monomers, oligomers, polymers, and crosslinkers for use in a variety of industries including Personal Care, Performance Chemicals and Pharma.